

Surface Micromachining for Transition-Edge Detectors

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Abstract— We are developing arrays of high-performance detectors based on superconducting transition-edge sensors (TES) for application in x-ray materials analysis as well as x-ray and sub-millimeter astronomy. In order to obtain the desired thermal time constants, as well as to provide thermal isolation from adjacent pixels, these arrays utilize micromachined thermal-isolation structures. Until recently, we have achieved thermal isolation of single-pixel devices by anisotropic wet etching of the entire Si wafer behind the pixel, leaving the detector supported by a thin Si_3N_4 membrane. Limitations of this technique make it undesirable for the fabrication of close-packed arrays. One possible means to achieve thermal isolation of close-packed arrays is surface micromachining. Here, a TES is fabricated on top of a Si_3N_4 membrane that is held above the substrate by a small number of support legs. Because the underlying wafer is not thinned or removed, the resulting detector chip is strong and requires no special handling. In this paper we describe the fabrication processes and present preliminary data on the properties of 64-pixel arrays of surface-micromachined TES x-ray detectors.

Index Terms— X-ray detectors, IR detectors, microcalorimeter, x-ray spectrometry

I. INTRODUCTION

TRANSITION-EDGE sensor (TES) x-ray microcalorimeters have been shown to have excellent performance for soft x-rays [1], [2], [3]. While the single-pixel detectors reported so far are useful for some applications, such as x-ray microanalysis, many of the potential applications require moderate- to large-scale arrays of pixels. Arrays are important not only for imaging applications such as those in astronomy, but for any application that would benefit from an increase in detector collection area such as x-ray microanalysis. Development of arrays of TES microcalorimeters requires some significant changes in the design and fabrication methods that we have used to make high-performance single pixels.

An x-ray microcalorimeter consists of an absorber to stop and thermalize the incident x-ray photons, a thermometer to measure the resulting temperature increase, and a weak thermal link to a cryogenic bath to enable a return to the base temperature after the measurement of the temperature excursion [4]. In a TES microcalorimeter, the thermometer consists of a superconductor that is temperature biased within its superconducting transition. In practice, most TES devices have been fabricated using proximity-coupled normal-superconductor bilayers as a thermometer, and much of the research to date has been on developing TES detectors using various bilayers such as Mo-Au

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[5], Mo-Cu [2], Ti-Au [6], and Ir-Au [7]. In this paper we will concentrate not on the absorbers and thermometers, but on the thermal isolation structures.

In general, the thermal isolation in most cryogenic thermal detectors is provided by membranes of low-stress Si_3N_4 formed by bulk micromachining [8]. In a typical process [9] the absorbers and bilayer films are deposited on a Si wafer that has been coated with a low-stress (Si rich) Si_3N_4 film. A window is opened in the silicon nitride on the back side of the wafer and all of the Si wafer that underlies the detector is removed by anisotropic Si etching using KOH. Further thermal isolation can be achieved by opening holes in the now free-standing Si_3N_4 membrane. Because of the crystallographic nature of the anisotropic KOH etch, severe constraints are placed on the possible shapes and placements of etch windows. A more modern replacement for the crystallographic etch is the deep-reactive ion etch (DRIE). This tool uses sidewall passivation to achieve vertical structures with very high aspect ratio, and is capable of removing the entire Si wafer from beneath the detector area.

The development of arrays of TES microcalorimeter x-ray detectors depends on the ability to form thermal isolation structures that are compact, robust and easy to fabricate. One approach is to use DRIE to form an array of isolated membranes separated by a thin grid of supporting Si. While this is possible, the resulting structure is delicate, and relatively little space is left for pixel wiring or additional thermal-crosstalk reduction structures. As an alternative approach, we are exploring the use of surface micromachining as a means to fabricate arrays of TES microcalorimeters. In this technique the Si_3N_4 is deposited on top of a thin sacrificial layer that is later removed from beneath the nitride. This leaves a membrane that is supported above the surface of the wafer by silicon nitride legs. Because the underlying Si wafer is not thinned or partially removed, the finished device is mechanically strong.

Surface-micromachining techniques have been applied by several other groups to create various thermal-isolation structures. X-ray microcalorimeters have been fabricated using an anisotropic front-side surface-micromachining process [10]. Here top-side windows are opened in the Si_3N_4 membrane that allow the Si underneath the pixel to be removed. Crystallographic constraints limit the applicability of this technique to close-packed arrays. Eriksson and co-workers [11] have developed surface-micromachined thermal-isolation structures for room-temperature IR detectors utilizing Si_3N_4 membranes and polyimide sacrificial layers. The polyimide sacrificial layers are removed by oxygen-plasma ashing, which limits this technique to relatively small ($\sim 50 \mu\text{m}$) pixel sizes. Free stand-

ing membranes have also been created using the XeF_2 gas phase chemical etch [12]. This etch can be used to remove under-pixel Si of relatively small pixels, or of open structures such as spider-web infrared bolometers [13]. Because this XeF_2 etch is isotropic, the depth and the lateral dimensions of an etched region are roughly the same, limiting the geometries that can be etched with this approach.

II. FABRICATION DETAILS

The basic concept of the surface micromachining methods we have employed is shown in figure 1. A sacrificial layer of poly-Si is deposited on an oxidized Si wafer and patterned into mesas. The membrane Si_3N_4 is then deposited onto the wafer, and the nitride is removed from appropriately designed windows. After completion of the TES processing, the sacrificial poly-Si is removed by a XeF_2 gas-phase chemical etch, leaving a silicon nitride table supported above the substrate by silicon nitride legs. We are able to etch large pixels by utilizing the unique features of the XeF_2 etch. First, the etch rates of SiO_2 , Si_3N_4 and photoresist in XeF_2 are orders of magnitude lower than that of Si [14]. Second, because the reactive species is molecular XeF_2 and not the ions used in a plasma process, they are unaffected by collisions with non-reactive surfaces and are able to etch into features much smaller than the gas mean-free-path. Thus by creating a sacrificial Si mesa that is encapsulated by oxide and nitride, we are able to build large (1 mm^2) membranes without removing any of the underlying substrate.

In our current fabrication process we use a $2 \mu\text{m}$ thick poly-Si sacrificial layer. This layer is deposited using a standard low-pressure chemical vapor deposition (LPCVD) technique. An important consideration is the slope of the poly-Si mesa walls. In order to obtain reliable wiring connections to the TES thermometer on top of the finished membrane, the support legs must have a sufficiently shallow slope. We have developed a sloped-sidewall poly-Si reactive-ion etch (RIE) utilizing a SF_6 and O_2 mixture that creates sidewall slopes of roughly 45° . Subsequent to mesa formation a $0.5 \mu\text{m}$ thick layer of low-stress Si_3N_4 is deposited using LPCVD. The conformal coatings created by LPCVD ensure that the support legs will have a thickness similar to that of the top surface membrane. The Mo/Cu bilayer, Cu sidewall passivation, and Bi absorber which comprise the microcalorimeter are then fabricated on top of the poly/nitride mesas by processes described elsewhere [9].

Our previous detectors utilized the thin (65 nm) Mo from the bilayer as a superconducting wiring interconnect. In this case something better is needed. Scanning electron microscope (SEM) images indicate that the thinning of sputtered metal films deposited on the sloped Si_3N_4 legs is significant. We have measured that the film thickness of the metal interconnects can be reduced to as little as 40 % of the original thickness in the steepest part of the leg slope. Because of this we have implemented an additional wiring layer. Experiments have shown that we can obtain reliable continuity with sputtered wiring layers as thin as 200 nm. To ensure large critical-current connection to the TES we are currently employing a 500 nm thick sputtered Nb wiring layer that is patterned using a lift-off process.

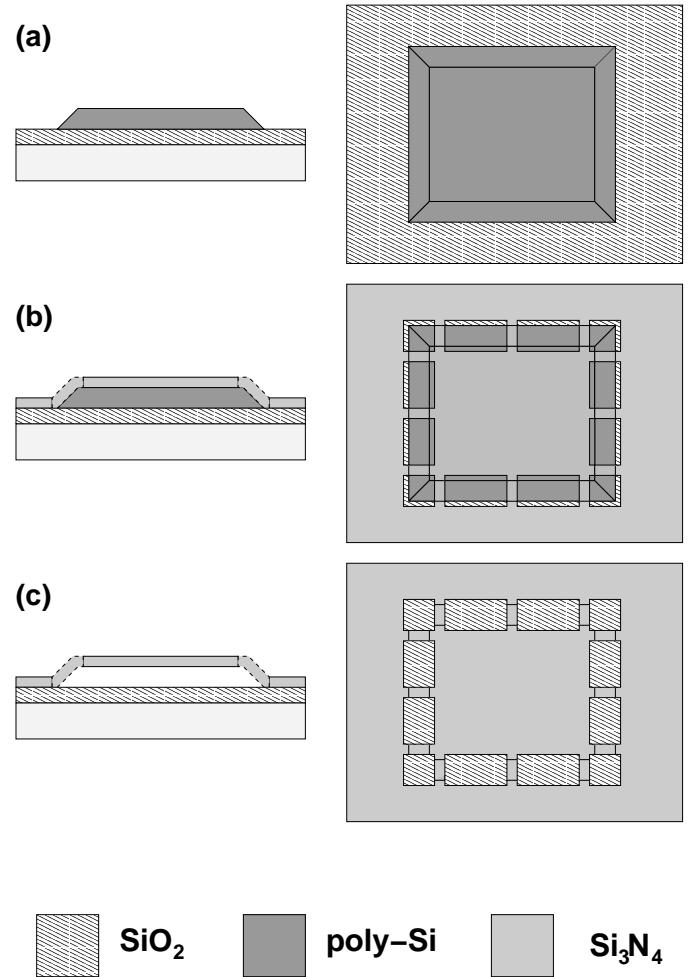


Fig. 1. Schematic processing for surface-micromachined detector configuration showing both cross sections and plan views. (a) The sacrificial poly-Si layer is deposited and patterned using a sloped-sidewall etching process. (b) The membrane Si_3N_4 is deposited and patterned with windows to allow the removal of the sacrificial layer. (c) The sacrificial layer is removed by a XeF_2 plasma-less dry etch, leaving a membrane supported by (in this example) 12 legs of Si_3N_4 .

One potential advantage of this surface-micromachined detector configuration is that the area underneath the pixel is potentially available for placing interconnect wiring or other ancillary structures. We have performed experiments to confirm the compatibility of the materials systems with this idea. Because we are currently using high-temperature (850 °C) deposition temperatures of Si_3N_4 to ensure good mechanical properties and conformal film coverage, the compatibility of standard superconducting wiring layers was unknown. We have deposited 300 nm of sputtered Nb, capped it with a low-temperature plasma-deposited SiO_2 , and then covered it with LPCVD poly-Si and Si_3N_4 . Measurements indicate that the high-temperature processing steps have reduced the superconducting transition temperature from 9.3 K to 7 K and increased the room temperature resistivity by a factor of two to $25 \mu\Omega\text{-cm}$, presumably because of oxygen incorporation from the adjacent SiO_2 films. The properties of this degraded Nb layer are still acceptable for use as a wiring layer. While we have not incorporated this buried Nb wiring layer in the devices described below, these measurements indicate that integration of such a layer will be

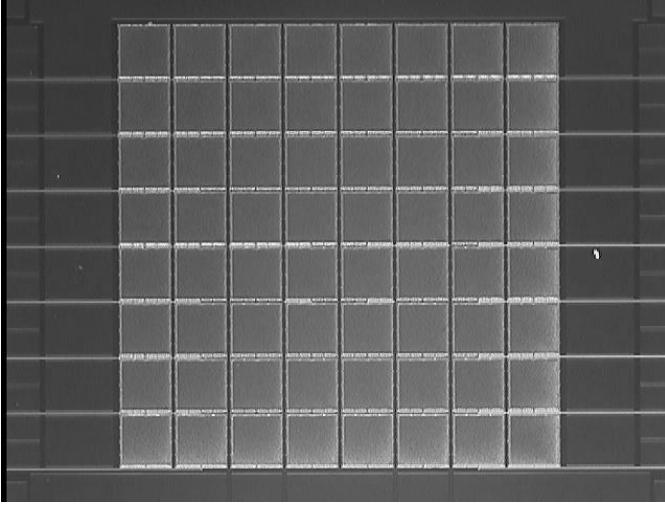


Fig. 2. Optical micrograph of an 8×8 array of TES x-ray microcalorimeters. In this chip the four central pixels as well as two columns of eight pixels each are wired to external pads and bias resistors. The pixels in this preliminary design are roughly $400 \mu\text{m}$ across and have a fill factor of greater than 75%.

straightforward.

After completion of all of the TES and wiring levels, we open windows in the Si_3N_4 with RIE to allow the removal of the poly-Si and to define the support legs. Because XeF_2 attacks Mo and Nb, we cover those structures with photoresist prior to the release etch. The release etch is carried out in a pulse-type XeF_2 reactor. Because the $\text{XeF}_2 - \text{Si}$ reaction is extremely exothermic it is important to minimize both the etch rate and the device heating. We accomplish both by diluting the XeF_2 with He. The high thermal conductivity of He gas at the operating pressure helps keep the etched structure in thermal equilibrium with the reactor walls. We admit a 1:1 mixture of XeF_2 and He into the reactor at a pressure of 300 Pa and allow it to etch the device for 20 seconds. The reactants are then pumped away and the process is repeated. It typically takes 50 such pulse cycles to completely remove the Si from underneath a 0.5 mm square membrane.

III. RESULTS AND CONCLUSIONS

We have fabricated working detectors using the surface micromachining techniques described above. Photographs of working completed detectors are shown in figures 2 through 4. While these detectors were fabricated as 8×8 arrays, our current test setup allows the measurement of only one pixel with each cooldown. Measurements of unreleased pixels (with the sacrificial layer intact) give us a bilayer T_c of 175 mK and normal-state resistances, R_n , of 13-15 m Ω . In the one released pixel measured to date we obtain similar numbers for both T_c and R_n (180 mK and 12 m Ω), indicating that heating or deformation due to the membrane release are not adversely affecting the bilayer films. The T_c obtained in this initial array is somewhat higher than the 120 mK goal. We believe this is due to drift in the bilayer deposition parameters, not to the surface micromachining processing: however, further measurements are needed.

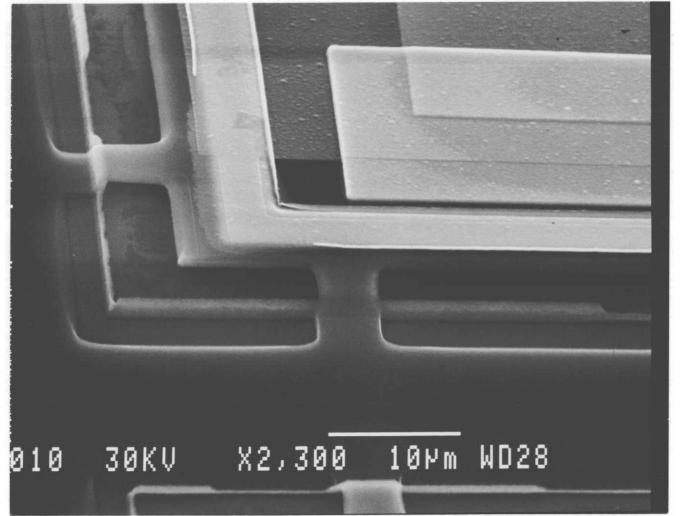


Fig. 3. Electron micrograph showing details of the Si_3N_4 support legs at the corner of the membrane table. Also visible is the Nb wiring and TES.

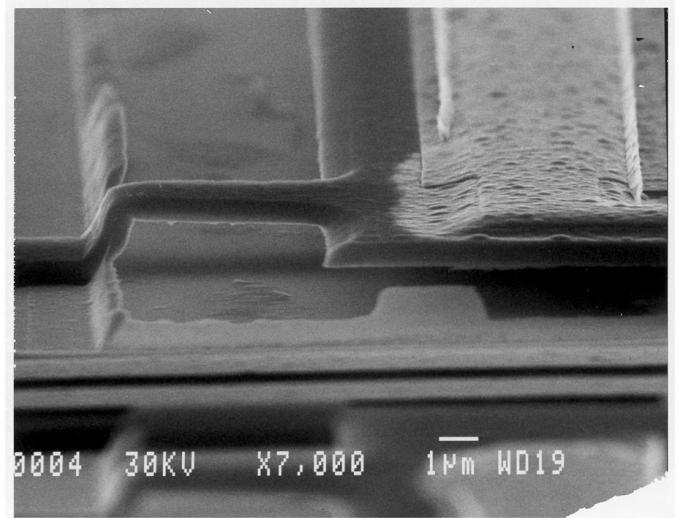


Fig. 4. Electron micrograph of one Si_3N_4 support leg. The large separation ($2 \mu\text{m}$) between the substrate and the membrane is clearly visible. Also visible is the Si_3N_4 "fence" near the legs. This is the remaining silicon nitride on the steep poly-Si sidewalls that is not removed when the Si_3N_4 windows are opened. It has no effect on the device.

When the detector is cooled to slightly below the transition temperature, voltage-biased, and illuminated with 5.9 keV x-rays from an ^{55}Fe source, we observe very fast current pulses with fall times of roughly 50 μsec . Because these pulses are measured with the bath temperature very near T_c , the pulse fall time is approximately the natural thermal time constant, τ . Using $C/G = \tau$ and a heat capacity, C , estimated from detector geometry and published materials properties we calculate an anomalously large value of the thermal conductance, G with values ranging from 20 nW/K to 40 nW/K. Estimates of G based on measurements of samples with similar geometries indicate that we should obtain values of roughly 6 nW/K at 170 mK. One possibility for obtaining an anomalously large thermal conductance is that the large mismatch in thermal ex-

pansion coefficient between the bilayer metals and the Si_3N_4 membrane causes the detector to deflect sufficiently to touch the substrate. Preliminary finite-element model calculations predict a deflection of 50 nm for the bilayer case and a 300 nm deflection when a 2 μm thick bismuth absorber is added. In both cases the predicted deflection is far less than the 2 μm spacing between the membrane and substrate. Additional causes, such as metal residue on the vertical portion of the Si_3N_4 legs, or incomplete removal of the sacrificial layer are currently under further investigation.

In conclusion, we have developed a surface micromachining process that is compatible with TES detector fabrication. We have used this process to fabricate 64-pixel arrays of detectors. Early measurements indicate that the bilayer metallization is unaffected by the additional processing, but that additional measurements are necessary to understand the anomalously large thermal conductance found in these structures.

REFERENCES

- [1] D. A. Wollman, S. W. Nam, Dale E Newbury, G. C. Hilton, K. D. Irwin, N. F. Bergren, S. Deiker, D. A. Rudman, and John M. Martinis, “Superconducting transition-edge-microcalorimeter x-ray spectrometer with 2eV energy resolution at 1.5 keV,” *Nucl. Instr. Meth. A*, vol. 444, pp. 145–150, 2000.
- [2] K. D. Irwin, G. C. Hilton, John M. Martinis, S. Deiker, N. F. Bergren, S. W. Nam, D. A. Rudman, and D. A. Wollman, “A Mo-Cu superconducting transition-edge microcalorimeter with 4.5 eV energy resolution at 6 keV,” *Nucl. Instr. Meth.*, vol. A444, pp. 184–187, 2000.
- [3] W. M. Bergmann Tiest, H. F. C. Hoevers, W. A. Mels, M. Ridder, M.P. Bruijn, P. A. J. de Korte, and M.E. Huber, “Performance of X-ray microcalorimeters with an energy resolution below 4.5 eV and 100 μs response time,” in *Low Temperature Detectors*, F. S Porter, Dan McCammon, Massimiliano Galeazzi, and Caroline K. Stahle, Eds. 2001, pp. 199–202, American Institute of Physics.
- [4] C. K. Stahle, D. McCammon, and K. D. Irwin, “Quantum Calorimetry,” *Physics Today*, vol. 52, pp. 32–37, August 1999.
- [5] F. M. Finkbeiner, T. C. Chen, S. Aslam, E. Figueroa-Feliciano, R. L. Kelley, M. Li, D. B. Mott, C. K. Stahle, and C. M. Stahle, “Fabrication of Superconducting Bilayer Transition Edge Thermometers and their Application for Spaceborne X-ray Microcalorimetry,” *IEEE Transactions on Applied Superconductivity*, vol. 9, pp. 2940–42, June 1999.
- [6] H. F. C. Hoevers, A. C. Bento, M. P. Bruijn, L. Gottardi, M. A. N. Korevaar, W. A. Mels, and P. A. J. de Korte, “Performance of a microcalorimeter with a superconducting transition edge thermometer,” *Nucl. Instrum. Meth. A*, vol. 444, pp. 192–195, 2000.
- [7] J. Hohne, M. Altmann, G. Angloher, P. Hettl, J. Jochum, T. Nussle, S. Pfnnur, J. Schnagl, M. L. Sarsa, S. Wanninger, and F. Von Feilitzsch, “High-resolution x-ray spectrometry using iridium-gold phase transition thermometers,” *X-ray Spectrometry*, vol. 28, pp. 396–398, September 1999.
- [8] J. J. Bock, D. Chen, P. D. Mauskopf, and A. E. Lange, “A Novel Bolometer for Infrared and Millimeter-wave Astrophysics,” *Space Science Reviews*, vol. 74, pp. 229–235, October 1995.
- [9] G. C. Hilton, John M. Martinis, K. D. Irwin, N. F. Bergren, D. A. Wollman, M. E. Huber, S. Deiker, and S. W. Nam, “Microfabricated Transition-Edge X-ray Detectors,” *IEEE Transactions on Applied Superconductivity*, vol. 11, pp. 739–742, March 2001.
- [10] T. Moreoka, K. Tanaka, Nakayama S, A. Nagata, K. Chinone, M. Ukiike, F. Hirayama, M Koyanagi, T. Mizuki, T. Hikosaka, U. Kawabe, and T. Nemoto, “Fabrication and characterization of superconducting X-ray calorimeters with transition edge sensors,” *IEEE Transactions on Applied Superconductivity*, vol. 11, pp. 751–754, March 2001.
- [11] Pontus Eriksson, Jan Y. Andersson, and Göran Stemme, “Thermal Characterization of Surface-Micromachined Silicon Nitride Membranes for Thermal Infrared Detectors,” *Journal of Microelectromechanical Systems*, vol. 6, pp. 55–61, Mar. 1997.
- [12] F. I. Chang, R. Yeh, G. Lin, P. B. Chu, E. Hoffman, E. J. J. Kruglick, and K. S. J. Pister, “Gas-phase Silicon Micromachining with Xenon Difluoride,” *Proc. SPIE*, vol. 2641, pp. 117–128, 1995.
- [13] J. M. Gildemeister, A. T. Lee, and P. L. Richards, “Monolithic arrays of absorber-coupled voltage-biased superconducting bolometers,” *Applied Physics Letters*, vol. 77, pp. 4040–4042, December 2000.
- [14] Kirt R. Williams and Richard S. Muller, “Etch Rates for Micromachining Processing,” *J. Microelectromechanical Systems*, vol. 5, pp. 256–269, December 1996.